Characterization and supply of raw materials in the Neanderthal groups of Prado Vargas Cave (Cornejo, Burgos, Spain)

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A systematic archaeological field survey has been undertaken in the area around Prado Vargas Cave (Cornejo, Burgos, Spain), which shows evidence of human occupation in the Middle Paleolithic. The aim of the study is to locate outcrops of raw materials which could have been used for the fabrication of tools by these Neanderthal groups. An archeological field survey of 46.6 km² in 94 different locations was undertaken, in which flint and other materials of archaeological and ethnographic interest were recovered. Different analytic techniques were employed (Fourier Transform Infrared Spectroscopy [FTIR], X-Ray Diffraction [XRD], and Inductively Coupled Plasma Mass Spectrometry [ICP-MS]) with the aim of typifying the lithic materials found in ten selected samples of flint on primary position in limestone and ten samples selected from flint on secondary position in clay. We have also undertaken the analysis of nine samples of archaeological flakes derived from the cave excavations. The flint samples were typified and the results of the data from the FTIR, XRD and ICP-MS were interpreted taking into account the similarity between samples of natural and archaeological origin, and the localization of possible areas of gathering of the lithic resources.

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1. Introduction

Archaeologists have always tried to find the sources of the raw materials used by the human groups of the past to make their lithic tools. This information becomes essential in order to formulate hypothesis about migratory phenomena, routes, colonization of other areas in the region, patterns of resources exploitation and the results of the data from the FTIR, XRD and ICP-MS were interpreted taking into account the similarity between samples of natural and archaeological origin, and the localization of possible areas of gathering of the lithic resources.

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between occupations and territory was found (Fernández, 1989; Orejas, 1991, 1995; San Miguel, 1992; Ruiz and Fernández, 1993; Goodchild, 1996; Conde et al., 2000; Chapa et al., 2003; García, 2004a,b; Moreno, 2004; Navazo, 2006; Iriarte et al. 2007; Marín, 2008; Ordono, 2008; Ordono and Arribalaba, 2009; Rodríguez, 2009; Navazo et al., 2010; Maximiano, 2011; Bergsvik and Skeates, 2012).

This present work focuses on the application of the methodology on surface survey as developed in the works of the aforementioned authors, adjusting it mainly to the location of flint outcrops on primary position (hosted in the limestone substratum), as well as on secondary position, that is, decontextualized from its original location — both in limestone materials as well as hosted into clays. It entails the covering of the widest possible area of terrain, obviously according to the factors that help or hinder the achievement of this goal (Mendez et al., 2004; Navazo, 2006; Rodríguez, 2009; Navazo et al., 2010). The geochemical study of flint can be used as a technique of characterization because during the processes of host rock dissolution the flint can be enriched with elements susceptible of being included into the flint genesis. On the other hand, even though the processes of silicification may be highly intense, usually there are remains of host rocks left, which are hard to value due to the standard mineralogical analyses. However, they can be indirectly inferred through their chemical composition (Bustillo et al., 2012).

In this present work we propose a characterization of the raw materials used by the Neandertal groups who occupied the cave of Prado Vargas. We develop a systematic surface survey with the aim of locating flint outcrops and perform geochemical and mineralogical analyses to determine their composition.

2. Regional setting

Prado Vargas Cave is located at the bottom of the left bank of the Ulemas River, upstream from Cornejo village, comprised within the Ojo Guareña karst in the municipality of Merindad de Sotoscueva (Fig. 1a, b). It is located within a large geological unit called Basque-Cantabrian Basin (Ramírez del Pozo, 1978; Jiménez, 1997; Tarríno, 2004; Tarríno et al., 2007), and is mainly constituted of materials from the Mesozoic Era. The depressed areas and the synclines are covered by Cenozoic sediments. This basin, comprising the west of Navarre, the Basque Country, the north of Burgos and Palencia provinces, and a great part of Cantabria, is characterized by the enormous power of its sedimentary successions, especially those of the Cretaceous (Del val et al., 2007).

Within the Basque-Cantabrian Basin we can differentiate three domains based on the structural criteria of age and the sedimentary characters of the outcropping rocks: from south-west to north-east these three domains are known as the North-Castilian Platform, the Navarre-Cantabrian Groove, and the Basque Arc. The Ojo Guareña karst is located in the second one, in the Navarre-Cantabrian Groove or domain. The Navarre-Cantabrian Groove correspondence to an area of important subsidence in the Mesozoic era, even though such a subsidence was compensated by a sedimentation which hindered the achievement of deep marine conditions.

From a structural point of view, and within the area under study, the materials in the northern part correspond to the Lower Cretaceous, with a high tectonic tranquility. In the southern part of the area under study, the materials correspond to the Upper Cretaceous and present a high tectonic activity — influenced by fold structures, faults, and important fractures (Ramírez del Pozo, 1978). The Upper Cretaceous is very well represented in the area under study, alternating between continental and marine events, from the Lower Cenomanian to the Middle-Upper Cenomanian and the Turonian, up to the Middle-Upper Coniacian where the Ojo Guareña karstic complex — to which the Prado Vargas Cave belongs — is developed. It is in this geological layer where the majority of the flint materials found are located (Fig. 2a).

From a geomorphological point of view, the slope relief stands out, a monoclinal structure of limestone materials more resistant than those found beneath them, which are of a weaker quality and which conform the scree (Ortega et al., 2013). The Somo hills develop to the north of the Cenomanian limestone escarpments, formed by sediments from the Lower Cretaceous, of terrigenous and siliceous character, producing a monoclinal series belonging to the Weald facies, the Aptian, and the Albian (Ramírez del Pozo, 1978). At the opposite side of the Coniacian limestone slopes there is a succession of fields of limestone pavements, colmated and active dolines of different sizes, as well as geomorphological structures which conform the exokarst. Likewise, towards the south of the Coniacian limestone slopes, a second slope of limestones and marls from the Middle-Upper Santonian is developed, as well as the hanging wall syncline of Mesa from the Santonian (Ortega et al., 2013).

From an archaeological point of view, Prado Vargas Cave is known from the works of exploration and cartography done in the karstic complex of Ojo Guareña by the speleological group Edelweiss (G.E.E, 1986), which in the decade of the 70s recovered a skull of Ursus spelaeus and other remains, placed at the Museum of Burgos. Trinidad Torres became interested in this cave and performed a dig in 1986 making several samplings in which remains of fauna and lithic material attributed to the Middle Paleolithic appeared (Torres et al., 1993; Navazo et al., 2005). Subsequently in 2006, Dr. Marta Navazo and her team from the University of Burgos began the work of digging in the cave, extending the previously dug area, and recovering lithic materials and fauna (Navazo et al., 2008). From the recovered materials at level four of the sampling called “Alpha” a dating through the technique of amino acid racemization in a horse premolar (Equus sp.) was performed, resulting in 46.4 BP (Navazo et al., 2005).

3. Material and methods

3.1. Survey

In order to design the archaeological survey correctly, a working plan was developed from the recommendations and approaches formulated by different authors (Ruiz, 1988; Ruiz and Fernández, 1993, 1996, 1997; Fernández, 1989; Vaquerizo et al., 1991; Bendala, 1992; Almagro and Benito-López, 1993; Renfrew and Bahn, 1993, 2008; Burillo, 1997; Cerrillo, 1997; Chapa et al., 2003; Cerrón et al., 2004; García, 2004a,b; Mendez et al., 2004; Moreno, 2004; Navazo, 2006; Navazo et al., 2008; Domínguez and García, 2007; Baena et al., 2008; Ripoll, 2010; Cerrato, 2011). These methodologies, originally focused on the search of locations with archaeological interest, have been adjusted to fit the main goal of the present work: the localization of flint outcrops, as well as to the results obtained in other related works in what pertains to the area of the gathering of resources (Tarríno, 2004; Navazo, 2006).

We start from the criterion of some of the authors (Higgs and Vita-Finzi, 1972; Davidson and Bailey, 1984) in relation to the radius of the optimum distance that the groups of hunter-gatherers could have inhabited, between 5 and 10 km (Lee, 1969); the calculation of distances based on the proportion of raw materials which appear in sites from the Middle Paleolithic in the southeast of France (Gamble, 2001); and the areas of gathering at different sites of the Basque Country (Tarríno, 2004). A radius of 5 km was delimited surrounding the site of Prado Vargas Cave, functioning as the center of the resulting circumference, an estimated area of 78.54 km².
Later, a work of documentation and bibliography was performed, in which the localization of the selected area was specified in the geological maps of the GMIS (Geological and Mining Institute of Spain), geological maps 1:50,000 MAGNA series 84 and 109 (Ramírez Del Pozo, 1978). The specific maps for the field work were also made at this stage; these were composed by the layers of the orthophotos from the PNOA 2005 NE, with a resolution of 25 pixels per cm, and by the layers of the cadastre from the municipality of Merindad de Sotoscueva, Espinosa de los Monteros, and Villarcayo de Merindad de Castilla la Vieja. Through the GIS GvSIG 1.11 and

Fig. 1. a: Survey area. b: General view of Sotoscueva Valley. Burgos. Spain.
Fig. 2. a: Geological map of MAGNA 84 and 109 (Ramírez del Pozo J and IGME, 1978), research area in white circle. b: Sector or units on survey area.
MIRAMON 6 software we performed the reclassification of the cadastral plots in order to make larger polygons so that they could be used as reference for the index cards of the materials. This resulted in five large units or sectors, with an easily recognizable lime between them on the terrain, thus avoiding an overlapping between different days of survey, and optimizing the invested time. The sectors or units corresponding with the northern area of the Conacian limestone slopes are the so-called U4 and U5 and are mainly composed of siliceous terrigenous materials, Aptian and Albian marine materials, and inclined plane screes, as well as of Quaternary materials deposited at the bottom of the Sotoscueva Valley. U1 and U2 correspond to the Coniacian limestone slopes, and material from more modern ages corresponds to the Upper Cretaceous, whose southern limit would be the Villamartín Fault — well delimiting the plots. Lastly, we have unit U3, which corresponds to the southern part of the selected area and is composed by materials from the Final Upper Cretaceous (Fig. 2b).

The chosen method for sampling was the systematic sampling of the whole occupied area, with a special emphasis on the geological layers susceptible of having silifications (Tarrino, 2004). The sectors labeled U4 and U5, which correspond to the northern area under study, are composed by siliceous terrigenous materials and do not contain silifications. We also registered all those elements of an anthropic origin and of any chronology, regardless of having a supposedly archaeological or ethnographic character (upright stones, walls, pit-houses, shepherds’ huts, charcoal kilns, etc.), for these are greatly helpful for evaluating the impact of the human groups in the territory.

In order to perform the transects on the terrain, we adapted the manner of proceeding to fit the terrain according to the contour line and the degree of vegetation coverage, a datum which was also registered on the daily survey index card, and which served us to evaluate the degree of difficulty in the localization of the materials. Other data such as the slopes, the karstification of the terrain, etc. could give us the level of mobility around the territory. These transects, as much as possible, were performed with a separation of 30–60 m, as the main objective was the detection of natural flint, both in primary and secondary position (Fig. 3). All sections of the limestone strata were revised, both natural and anthropic, as well as the bottoms of the ravines, since these are ideal places to find rounded materials. We recovered samples from all locations, and selected those materials that best represented the characteristics of flint. Those samples which were not found in an outcrop of the material were also recovered, being thus specified in the collection’s index card.

3.2. Analysis

The techniques employed for the characterization of flint included in this research were the Inductively Coupled Plasma Mass Spectrometry (ICP-MS), performed in the laboratories of the National Research Center on Human Evolution (CENIEH) in Burgos, Spain, and the Fourier Transform Infrared Spectroscopy (FTIR) and the X-Ray Diffraction (XRD), performed at the Department of Mineralogy and Petrology of the Faculty of Science and Technology and in the laboratory of the Advanced Research Facilities (SGiKer) at the University of the Basque Country (UPV/EHU) in Leioa (Vizcaya, Spain). Out of the total number of samples gathered during the survey only the most representative were selected, both on primary position (located in the place where they were generated) and on secondary position (materials which were decontextualized from their place of origin). Likewise, some samples of flakes and lithic archaeological remains originating from the digging in the site of Prado Vargas Cave were the object of geochemical study, with the goal of connecting the results obtained from natural flint to those from the archaeological remains.

The samples to be analyzed had to contain enough non-altered flint and be representative of the outcrop. The external surface was removed, the core was cut into smaller pieces and the obtained fragments were given and acid and ultrasonic bath to remove

![Fig. 1. Map with tracks within and outside delimited survey area.](image-url)
impurities. Later the fragments were ground in a wolfram disc mill and then centrifuged at 1000 rev/min for 30 s. For the infrared spectra of the samples in a Jasco FT/IR 610 spectrometer we needed to prepare KBr pellets. The spectra were then filed in a text format and processed through the Kaleida software. The X-Ray Diffraction was performed in a Phillips X’Pert diffractometer. The diffractograms were performed with an interval from 10° to 90° in 2θ, at a 0.026° interval, and with a time interval exposure of 49.5 s. The ICP-AES measurements were performed using a Perkin Elmer Optima 5300 DV inductively coupled plasma atomic emission spectrometer. The microwave digestion procedures were performed with a CEM MARS closed vessel acid digestion system. All the samples were measured three times and the mean value of the composition was taken as representative of the whole surveyed area. 16 chemical elements were analyzed in the flint. Concentration values are expressed in μg of chemical element per gram of solid sample (μg g⁻¹).

4. Results

4.1. Survey

The field work took place over a period of 33 days. A total of 313 km of transects were conducted in 46.26 km². 94 flint locations, 10 archaeological flint artifacts, 3 archaeological quartzite artifacts and 57 places of interest (upright stones, pit-houses, charcoal kilns) were found (Figs. 4 and 5a–c). All the information was collected on the field work index cards which had been previously elaborated. During the survey phase, modifications were made to the transects so that the employed methodology could be more productive at field work, adjusting it to the geological and geomorphological terrain characteristics. The materials which supposedly showed signs of intentional knapping were georeferentiated separately (denominated “Archaeological Flint” in the site plan).

4.2. Analysis

The analysis of 20 samples of natural flint was performed. 10 of them were on primary position (hosted in the limestone stratum), and the other 10 on secondary position (decontextualized — outside their original position — and in clay). Besides, other 9 samples of archaeological material recovered from the excavations in Prado Vargas Cave were analyzed. In order to select the natural samples to be analyzed, we considered the quality of flint, the available quantity, and the fact that they had to be representative of the whole surveyed area.

4.2.1. Infrared spectra

On all infrared spectra, we observed the characteristic absorption bands from the SiO₂ quartz, between 1100 and 500 cm⁻¹ (Fig. 6a, b), with an intense broadband located between 1110 and
1188 cm⁻¹. We have noticed two main differences between the samples on primary position (hosted in limestone), those on secondary, and the archaeological samples (Fig. 7a, b). The maximum of weak absorption located around 1385 cm⁻¹ was only observed in flint spectra on primary position, whereas in the spectra on the secondary position and in the archaeological samples the spectra are absent. However, in the maximum of absorption observed at 555 cm⁻¹ the opposite occurred, it was present on the secondary and archaeological samples but absent on the primary ones. In Table 1, in the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) results, in the values obtained for calcite, a decrease can be observed in the samples on secondary position and in some of the archaeological ones, thus relating it to the variation in intensity in the infrared spectra mentioned above.

Several authors have tried to tentatively associate the maximum of absorption located at 1385 cm⁻¹ to different anionic groups, such as the CO₃⁻ carbonate (1410 cm⁻¹) (Stuart, 2004) or the NO₃⁻ nitrate (1410–1340 cm⁻¹). One of these authors (Coates, 2000) to different anionic groups, such as the CO₃⁻ carbonate (1410 cm⁻¹) (Stuart, 2004) or the NO₃⁻ nitrate (1410–1340 cm⁻¹). One of these authors (Coates, 2000) maintains within the same value table for inorganic groups the same groups already mentioned.

We can establish, between the obtained spectra of the samples on primary position on the one hand, and the samples on secondary position and the archaeological ones on the other, a relationship between the material employed for making the lithic tools and the flint on secondary position, a relation already formulated by M. Navazo for the Sierra de Atapuerca (Navazo, 2006), which states that a 98% of the lithic tools were made of flint from secondary deposits. However, in the band located at 555 cm⁻¹ the opposite occurs. There appears an intensity band in the archaeological samples which does not appear in the natural ones, although showing a lower intensity — the causes of this intensity in the spectra could not be determined.

Certain authors (Pinzaru et al., 2008; Olivares et al., 2009, 2012, 2013; Parish, 2009, 2010, 2011; Hassler et al., 2013) consider that infrared spectroscopy can offer good opportunities for examining the lithic materials coming from different areas. The technique is faster and it seems to have a lower economic cost with respect to other techniques. However, it is a technique which shows limitations and cannot stand alone in order to characterize the materials of an area, although it can be useful for the comparison of different areas.

4.2.2. X-Ray Diffraction

Diffraction patterns were detected for the flint sample on primary position (702) and in an archaeological sample (731). The diffraction patterns corresponded to the mineral SiO₂, and no additional maximums were observed which could have indicated the presence of any other mineral in the samples (Fig. 8). Besides, the X-Ray Diffraction did not show significant differences in the diffractograms between the primary and the archaeological flint samples.

4.2.3. Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

In the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) 29 samples were analyzed — all those selected. All samples were measured three times and we took the mean value of the composition as representative of the sample. We studied 16 elements in the flint, as shown in Table 1. The high number of analyzed elements made the use of multivariate statistical tools for the study of the samples necessary. Dr. Álvaro Colina, from the Area of Analytical Chemistry at the Science Faculty of the University of Burgos, performed an analysis of the data by means of main components through the R statistical software.

Initially, a study was performed without autoscaling the data. Fig. 5a shows the representation of the scores and the loadings of
the first and second main components, in which the numbering corresponds to the samples. The loadings of each element are represented by the red arrows.

The non-standardization of the data implies that the elements which appear in a high quantity (Calcium and Aluminium) will have a greater weight in the main components. The variation in the quantity of Calcium is the major influential factor on the first main component, being the quantity of Aluminium the most influential factor on the second main component.

The samples on primary position (in limestone) had, in general, higher values of calcium than those on secondary position (in clay), although some of the samples had a lower content of Calcium than the one expected. This can be better observed in Fig. 9b, which shows the representation of only two scores, denoting in red the samples on primary position in limestone, in black the samples on secondary position in clay, and in green the samples of an archaeological origin.

The samples in clay had very low or null concentrations of Calcium, as it was to be expected, although samples 2 (702), 3 (703), and 10 (710) also had a very low concentration of Calcium in spite of being in limestone. The archaeological samples seemed to have a significant quantity of Calcium, even though samples 21 (721), 28 (728), and 29 (729) showed very low quantities of Calcium.

It should be highlighted that sample 30 (730) showed clear differences in composition with respect to the rest of the samples, although this could be better seen when the main components were performed with the autoscaled data. Thus, both the influence of the major components and that of the minor flint components could be observed (see loadings in Fig. 9c).

The standardization of the data (Fig. 9c) caused the Calcium and the Strontium to have a clear influence on the second main component. Likewise, the influence of Lanthanum and Lithium could be observed. Contrariwise (negative weigh), this component had also the influence of Zinc and Sodium. The first main component was influenced by the rest of the elements with a quite similar representativeness. After the autoscaling of the data, Fig. 9b, it could be observed that there was no relation between the standardized composition and the hosting in which the flint was located. No significant groupings were appreciated; standing out — as we have said earlier — the clear difference of sample 30.

After performing the statistic treatment of the obtained values with the ICP-MS, we observed that there were samples with an apparent similarity in spite of having different positions; sample 710 (10) on primary position in limestone, and sample 717 (17) on secondary position in clay, georeferenced as being very close to each other and at a distance of 2833 m and 3052 m on a straight line from the cave, in an area very near the main entrances of the karst of Ojo Guareña and with well-known archaeological sites (Fig. 9d). Besides, these samples also had a similar composition with respect to the flake archaeological samples 721 (21), 727 (27), and 729 (29). Archaeological sample 723 was also correlated with other two samples on secondary position in clay, these were samples 714 (14) and 716 (16), located at 1900 m and 1281 m respectively. In view of these samples, we can affirm that the majority of the archaeological samples could have had a local origin.

One of the samples, 730, is not related to any of those analyzed, being clearly separated from the rest, which makes the possibility of considering that this could be a material from other areas very feasible. M. Navazo (Navazo et al., 2005) related two lithic tools (quartzite) recovered from the cave site with the possibility of having an allochthonous origin. The analysis of this sample (730) could provide information about the movements of the Neanderthal human groups, supposing that it could be related to other areas where these materials were also characterized.

The locations where the samples of natural flint were found — which coincided with the archaeological ones — have noteworthy spatial and geomorphological characteristics: of territory control, abundance of surface flint, and available places of refuge (caves and shelters) (Fig. 10a,b). Moreover, according to the distances from these places to Prado Vargas Cave, we can confirm the local
Fig. 6. a, b: General spectra of all samples. All of them show typical intensity of SiO₂.
Fig. 7. a, b: Natural Flint on primary position samples 701–710, spectral fraction and archaeological samples. Differences among samples shown.
character of some of the materials obtained in the dig of the site and the mobility of the groups of hunter-gatherers as mentioned by some authors (Vita-Finzi and Higgs, 1970; Higgs and Vita-Finzi, 1972; Davidson and Bailey, 1984; Gamble, 2001).

After observing the similarity in the data of some of the natural flint samples to the data obtained in some of the archaeological samples, we verified that they correspond to three geographical locations having the three following conditions: abundance of raw materials, proximity to Prado Vargas Cave, and command of the territory. Geochemical results could be used in order to localize potential places with archaeological interest, developing selective surveys according to the geolocations of the natural materials used and their relation to the archaeological materials. A future work on these areas could offer new data to better understand the use of the space by these human groups.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) could provide very useful information at an elementary level (Tarríno, 2004; Navazo, 2006; Brandl et al., 2011), but it requires a long, arduous, and expensive treatment of the samples, as well as a statistical treatment of the data.

5. Conclusions
- The surface survey — the location of the outcrops of primary materials — provided information in order to understand the relationships between the Neanderthal human groups and the
environment. The data obtained concerning the sampling effort contributed to the planning of the surveys on the terrain.
- The characterization of the lithic material used by the Neanderthal groups who occupied Prado Vargas Cave in Cornejo (Burgos, Spain) has been achieved.
- The analysis of the results on the geochemical and mineralogical studies has been inconclusive with respect to the existence of clear markers that may separate these materials from other nearby geographical areas, or that may form groups of differentiated materials.
- By means of the Infrared Spectroscopy technique we have observed differences between the spectra of natural materials and the archaeological materials. However, it has not been possible to accurately identify the origin of the bands (around 1.385 cm\(^{-1}\) and 550 cm\(^{-1}\)) that may differentiate such spectra. The results obtained in the X-Ray Diffraction have not provided any relevant information on these differences obtained in the Infrared Spectroscopy.
- The statistical treatment of the values obtained by means of the ICP-MS indicates the existence of an apparent similarity between the samples of natural flint on different positions in relation to those of the archaeological samples. In view of these samples we can affirm that the majority of the archaeological samples, except for sample 730, could have a local origin.
- The locations where the samples of natural flint are found, which coincide with the analytic results of the archaeological samples analyzed, have noteworthy spatial and geomorphological characteristics: of territory control, abundance of surface flint, and available places of refuge (caves and shelters).
- According to the results of the geochemical analyses that the flint samples provide and the similarities that these results have to those of the archaeological samples, we can project a geographical location. This information could be used to perform selective surveys in order to locate places with potential archaeological interest.

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